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The NIF point design uses a five-layer capsule to modify the x-ray absorption in order to achieve optimized shock timing. A stepped copper dopant design defines the layer structure. The production of the capsule involves pyrolysis to remove the inner plastic mandrel. Cu atoms diffuse radially and azimuthally throughout the capsule during pyrolysis. This diffusion significantly diminishes the capsule's performance during implosion. Thermal and coated oxide barrier layers employed between layers mitigate the diffusion of copper during the mandrel removal process. The copper atoms do not diffuse through this barrier during pyrolysis. A capsule fabrication method that produces a capsule with a thin oxide layer will be discussed.

I. INTRODUCTION

Beryllium is one of the preferred ablators for achieving ignition at the National Ignition Facility (NIF) because of its hydrodynamic stability, low opacity, high thermal conductivity, and high tensile strength.[1] Fabrication of these capsules involves sputtering beryllium onto plastic mandrels; the specific coating process has been defined elsewhere.[2–4]

A stepped copper dopant profile is introduced to modify the x-ray absorption (Fig. 1). The copper prevents x-ray preheating of the fuel, suppresses Rayleigh-Taylor instability growth, and helps control shock timing. The capsule is manufactured by sputter coating beryllium and copper onto a Carbon-Hydrogen polymer (CH) mandrel. This fabrication method produces capsules with distinct copper steps. A hole is drilled in the capsules and the mandrel is removed at 425°C in an oxidizing environment. After pyrolysis, the copper has diffused heterogeneously throughout the capsule[5] (Fig. 2). Radial copper diffusion has the potential to be viable in a target design, provided that it is repeatable and quantified. However, the copper diffusion is observed to be non-uniform azimuthally as well as radially. This cannot be effectively modeled and would not be expected to efficiently modulate the timing of the implosion. The heterogeneous copper diffusion is thought to be caused by grain structure variations in sputtered beryllium.[5,6] The addition of thin metal oxide layers was proposed as a possible method to halt copper diffusion during the heated mandrel removal process. Other disciplines have effectively employed oxide layers to halt diffusion.[7,8] Our work focuses on the successful development of an *in-situ* oxide layer to halt copper diffusion.

II. EXPERIMENTATION AND RESULTS

The beryllium coaters used in these experiments all have the same general system configuration. They employ a single 3.3 cm Mini-MAK gun located approximately 2.5 cm above a rotating pan, which contains mandrels made of Glow Discharge Polymer (GDP). The gun operates at 50 W and the pan is biased. An anode ring operating at 45 V is located 6.5 mm above the pan. The copper gun is located at an angle to align it with the pan and runs at 4–15 W, depending on the desired copper concentration. For these experiments, our initial tests focused

on the deposition of a copper doped layer of beryllium sandwiched between two pure beryllium layers. An oxide layer was placed at one of the two resulting doped-undoped interfaces. The capsule was drilled and pyrolized at 425°C. We then compared the treated and untreated layer interfaces in the capsule.

Our first experiment deposited a thin Al_2O_3 layer deposited by Atomic Layer Deposition (ALD). ALD was selected because this technique has the advantages of depositing an extremely conformal film thin film layer. ALD is a well-established method for depositing copper diffusion barrier layers at less than 5 nm in thickness. **Error! Reference source not found.Error! Reference source not found.**,10

We coated various thicknesses, from 1 to 8 nm of alumina, at the interface. We included two control samples, which were thermally treated and a sample, which was held alongside the samples and received no treatment. Qualitatively, none of the treated samples demonstrated copper diffusion through the oxide layer. In Fig. 3, a backscattered electron image (BSE) shows the typical qualitative result observed. We also observed delamination at the oxide interface on many of the samples (Fig. 3 inset).

Additionally, copper did not appear to diffuse through the thermally treated layers of the control samples. This implied that a thermally grown oxide layer could be effective in halting copper diffusion. Other research supports this observation.[11,12] The production of a beryllium oxide layer is desirable because it is less time consuming and does not introduce aluminum into the sample matrix.

To produce the thermally grown oxide layer, we took samples from the sputter coater and treated them at 250°C for 6 h. We then returned the samples to the sputter coater and continued

the coating process. Analysis using BSE and Electron dispersive spectroscopy (EDS) both showed diffusion through the non-treated interface, and no diffusion through the oxide layer (Fig. 4).

The layers were observed in a polished cross section of the capsule. To prepare a polished cross section, a capsule was drilled and the mandrel was thermally removed. The sample was then potted in epoxy and dry polished to approximately half of a capsule. There is no support on the interior of the capsule during this process; this stresses the capsule walls. The layers within the capsule were often delaminated from each other when characterized, but this may have been induced by the sample preparation method. To observe the capsule wall without the introduction of polishing stress, we prepared a samples using Focused Ion Beam (FIB) cross-sectioning. The FIB results did show some evidence of weaker adhesion along the layer interface, but it was not as extreme as the sample prepared with polished cross section (Fig. 5).

Another method to produce an oxide layer was tested. During sputtering, oxygen was introduced into the sputter chamber for a set period of time. The coating was continuous during this process. Introducing a blend of 20% oxygen in argon into the chamber for 3 min immediately before a change in copper dopant concentration halted the copper diffusion (Fig. 6). Further analysis was done to inspect for delamination. The FIB cross sectioning capabilities only allow for 30-40 μm depth cuts into a beryllium capsule. To inspect a capsule that was coated to 120 μm, we fabricated a polished cross-section. We then used the ion beam to cut out a section of the capsule under a noted area of delamination, and inspected this area at the deepest section of the sectioning (Fig. 7), 40 μm under the polished defect, we did not observe delamination (Fig. 8). It should be noted that the area inspected by the FIB is much smaller and this inspection

amounted to 0.5% (30 μm) of the 5.65 mm circumference. Several additional randomly selected areas were analyzed with FIB, with similar results. Nonetheless, it should be noted that this inspection technique is not an effective way to characterize the entire layer interface within the capsule. Large voids along the interface would significantly affect shock timing, and a non-destructive radiography technique for complete capsule analysis is being developed.

Typical layers were also characterized using Rutherford backscattering (RBS) and Auger electron spectroscopy (AES). Combining these two techniques gives the details of the oxide composition and thickness. Additional studies were done on layers produced using oxide barriers,[13] and the results of those experiments show that the BeO layer is 7–9 nm thick when a 3 min *in-situ* oxide treatment is incorporated into the coating.

III. CONCLUSIONS AND FUTURE WORK

Without inhibition, Cu diffuses by several microns on average within the capsule layer structure. This diffusion can be halted by the use of a non-permeable oxide layer. An *in-situ* oxide layer has been developed and can produce capsules that meet the layer design specifications as desired by NIF experimentalists in the current design revision. The oxide layer may have some issues with delamination, but it is not clear whether this delamination occurs during sample preparation for inspection or if a significant amount of voids is present in the capsule as-coated. More studies are being conducted to verify that there is not a prohibitive number of voids in the capsule wall.

Acknowledgment

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FIGURE CAPTIONS

Fig. 1. NIF point design for beryllium ablator has five stepped layers with varied Cu at%.

Fig. 2. SEM backscattered electron (BSE) images of polished cross-section (A) pre-pyro (B) post-pyro.

Fig. 3. Left hand image of SEM backscattered electron (BSE) of polished cross-section with oxide layer post-pyro. Diffusion (observed as gradual intensity transition) can be seen at the non treated interface, but is not visible at the oxide interface, which distinctly changes in intensity. Right hand image shows ALD Al2O3 sample with significant delamination at the oxide interface.

Fig. 4. (a) BSE image of polished cross-section with oxide layer post-pyro. (b) EDS of the same capsule. Both indicate that copper diffusion was halted at the thermally induced oxide interface.

Fig. 5. Cross section preparation: a sample prepared with polished cross section (A) showed delamination across the interface. FIB cross-sectioning (B) showed much smaller voids along the interface boundary.

Fig. 6. BSE images of a polished cross-sectioned capsule with doped layers representing the NIF Be capsule design specifications.

Fig. 7. Diagram of sample FIB sample preparation for layer inspection.

Fig. 8. SEM inspection of FIB sectioned capsule beneath area of delamination from polished cross section. Curtaining, an artifact from sample preparation, caused the horizontal lines across the FIB image.

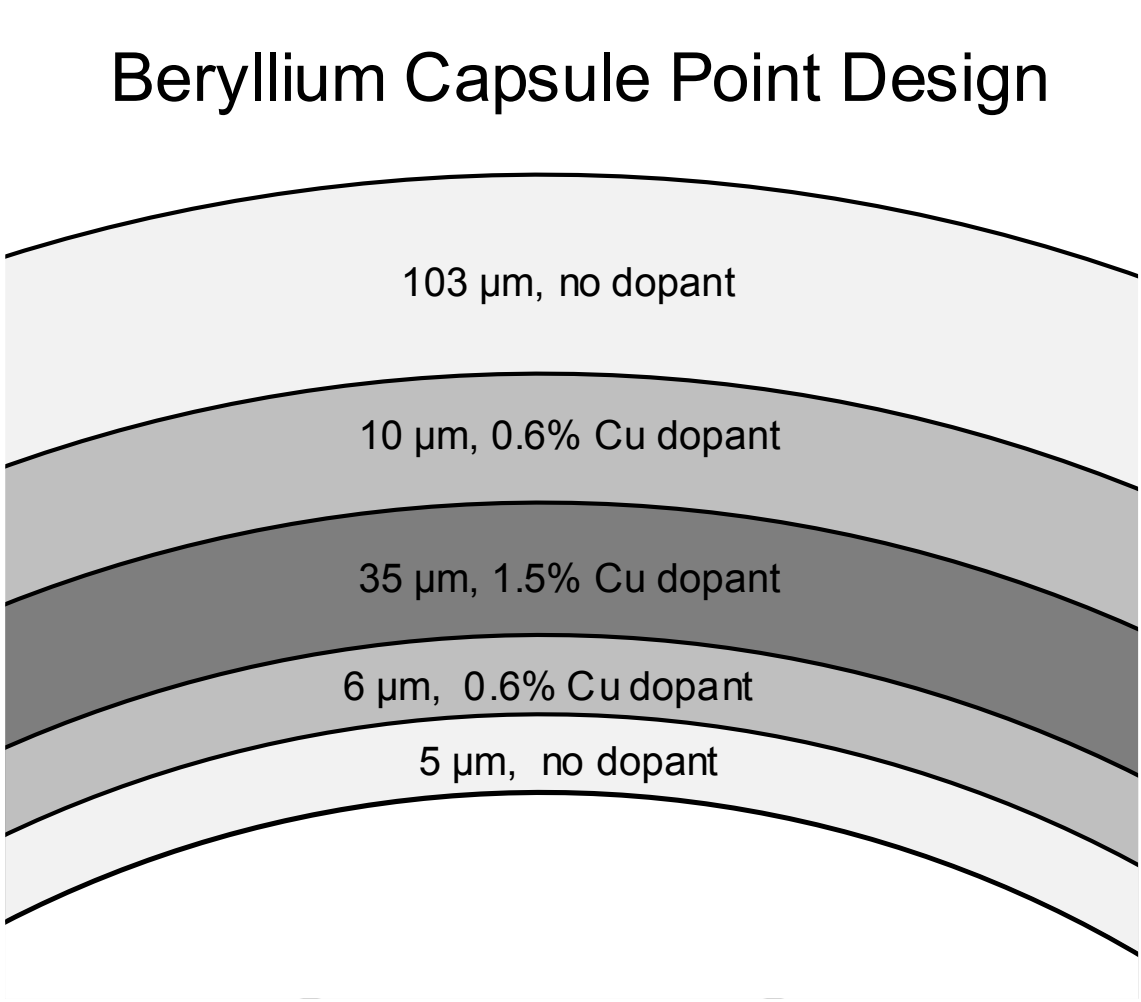

Beryllium Capsule Point Design
103 μm, no dopant
10 μm, 0.6% Cu dopant
35 μm, 1.5% Cu dopant
6 μm, 0.6% Cu dopant
5 μm, no dopant

Figure 1.

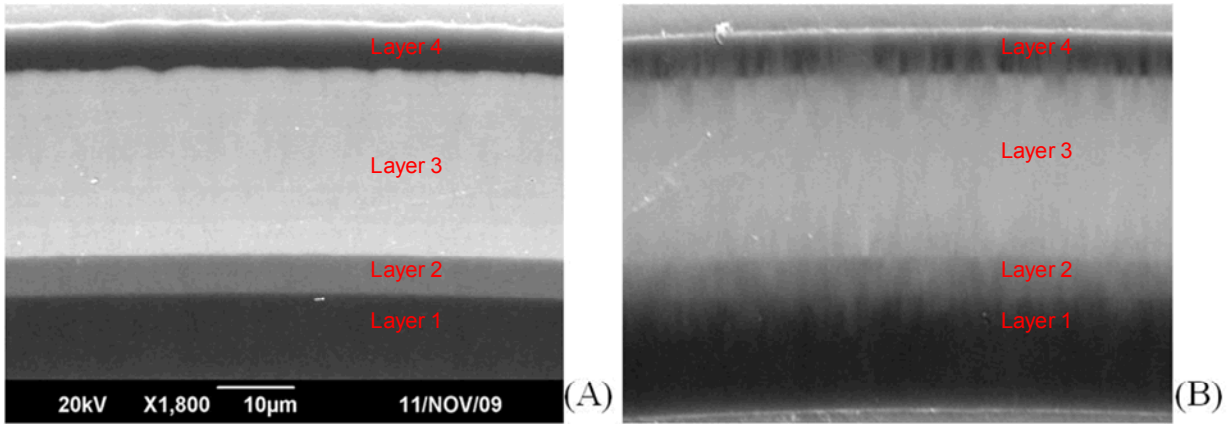

Layer 4
Layer 3
Layer 2
Layer 1
20kV X1,800 10µm 11/NOV/09
(A)
Layer 4
Layer 3
Layer 2
Layer 1
(B)

Figure 2.

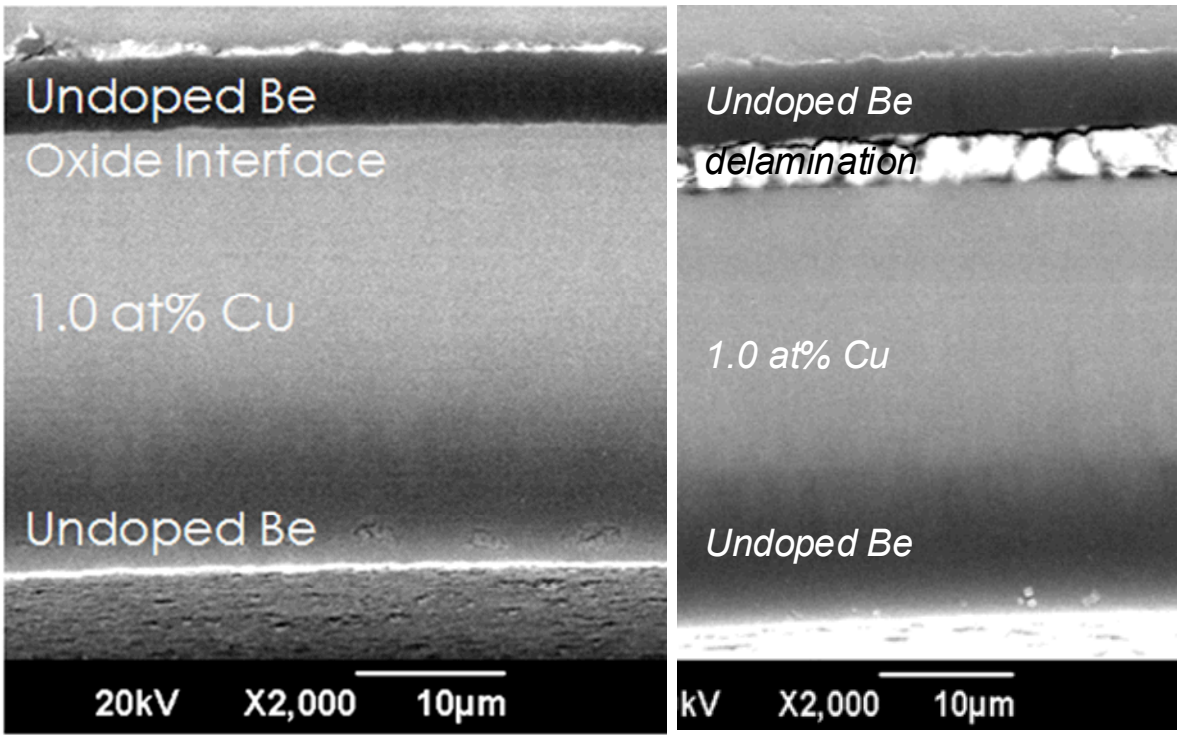

Undoped Be
Oxide Interface
1.0 at% Cu
Undoped Be
20kV X2,000 10µm
Undoped Be
delamination
1.0 at% Cu
Undoped Be
kV X2,000 10µm

Figure 3.

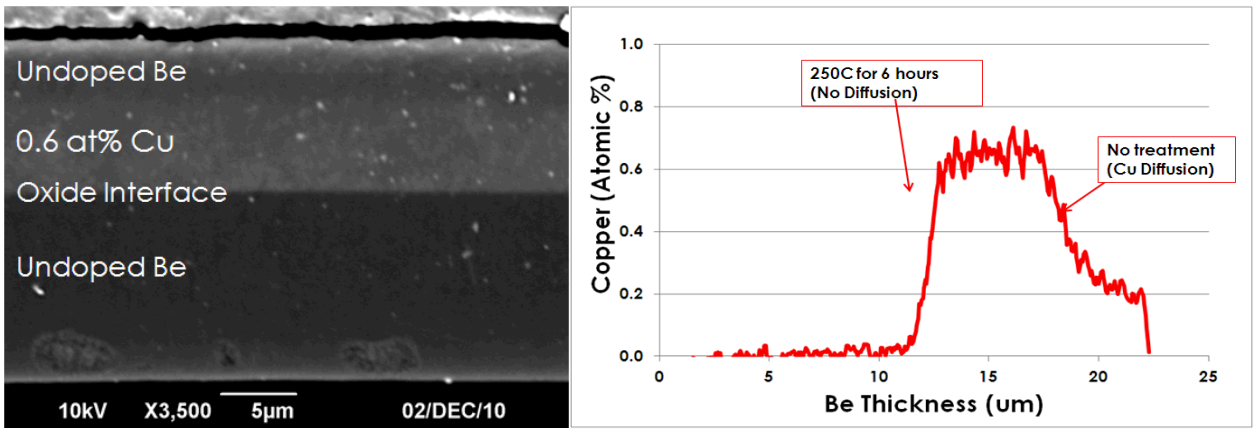
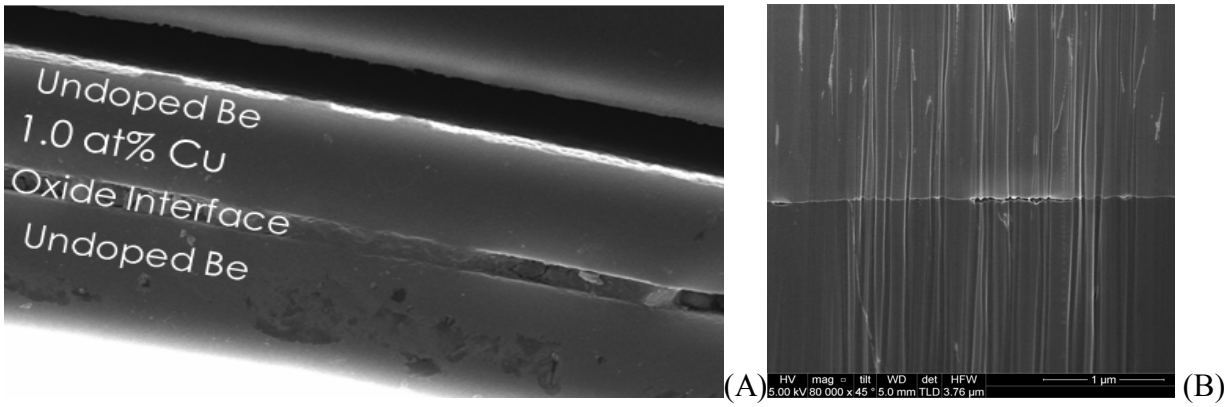

Undoped Be
0.6 at% Cu
Oxide Interface
Undoped Be
10kV X3,500 5µm 02/DEC/10
250C for 6 hours
(No Diffusion)
No treatment
(Cu Diffusion)
Copper (Atomic %)
1.0
0.8
0.6
0.4
0.2
0.0
0
5
10
15
20
25
Be Thickness (um)

Figure 4.


Undoped Be
1.0 at% Cu
Oxide Interface
Undoped Be
HV mag tilt WD det HFW
5.00 kV 80 000 x 45 ° 5.0 mm TLD 3.76 µm
1 µm

(A) (B)

Fig. 5.

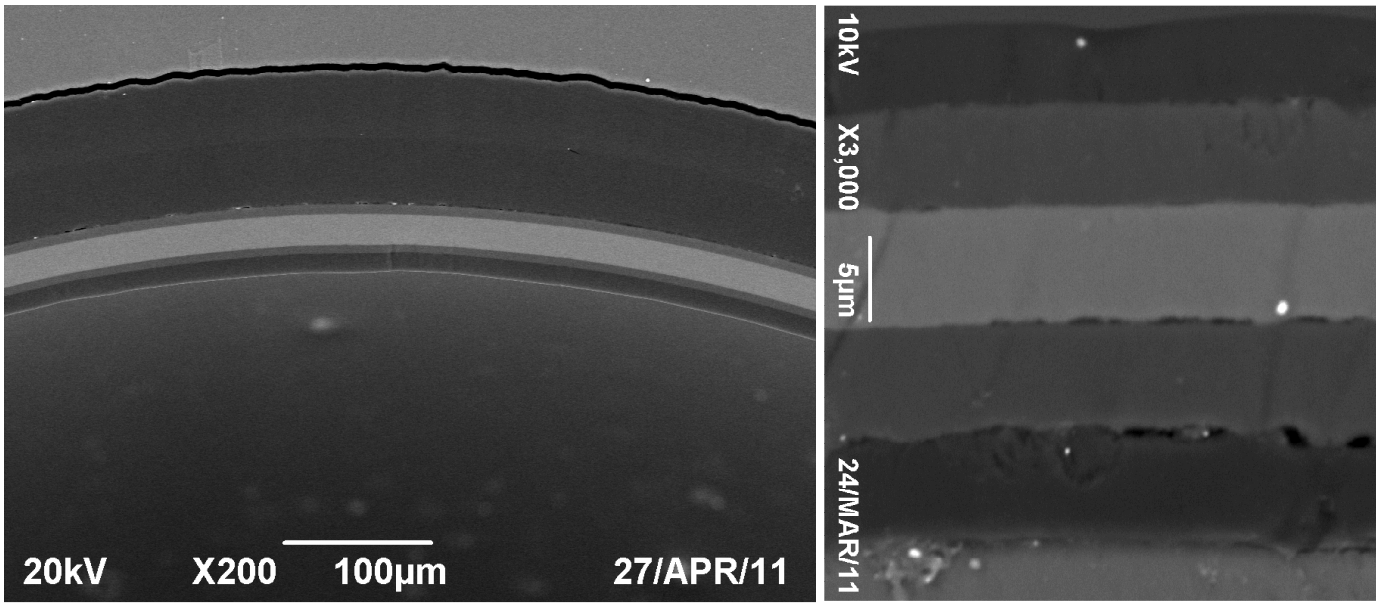


Fig. 6.

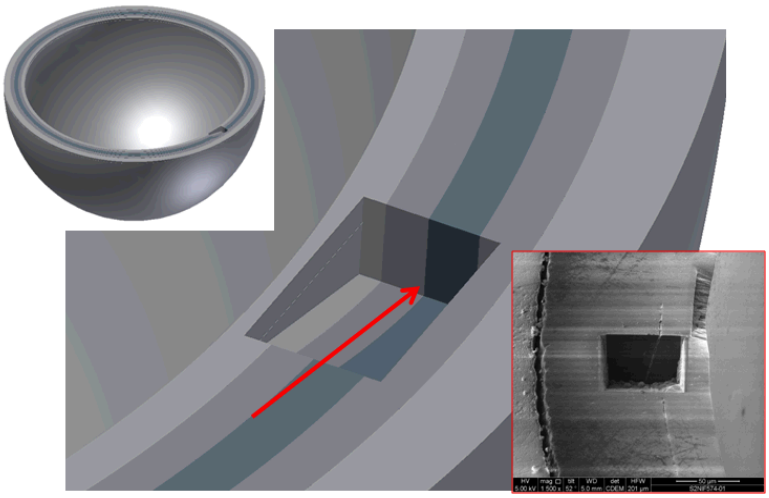


Fig. 7.

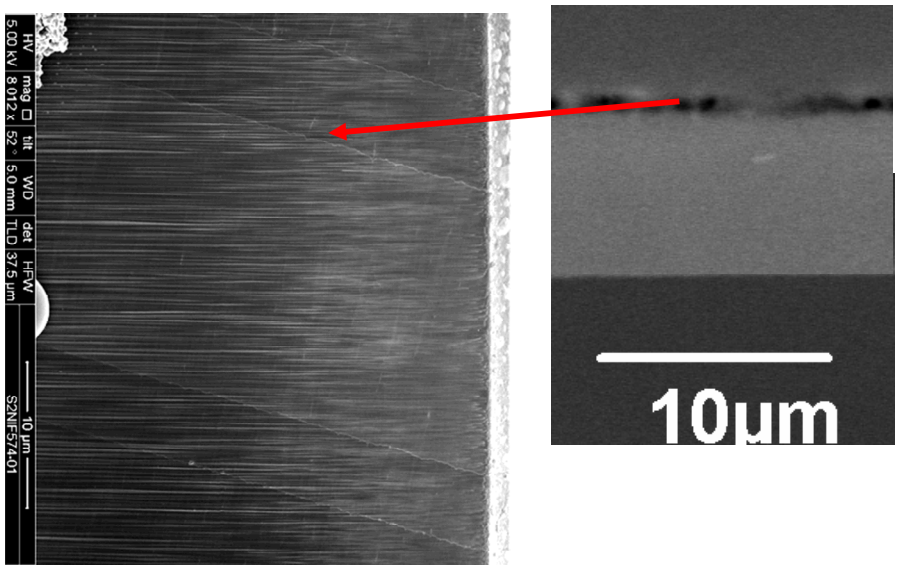

HV
5.00 kV
mag
8 012 x
tilt
52 °
WD
5.0 mm
det
TLD
HFW
37.5 µm
10 µm
S2NF574-01
10µm

Fig. 8.